# Segregation in an Inverted Loopseal for Use in Advanced Pressurized Fluid Bed Applications

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## Introduction

A Loopseal is commonly used in Circulating Fluid Bed (CFB) combustors in order to recirculate flyash from a low-pressure cyclone into a higher-pressure combustor. In an Advanced Pressurized Fluid Bed (APFBC) application, such as that described by Adams et al. (1999), a non-mechanical valve (an N-valve or loopseal) can be used to provide the seal between the high-pressure, spouted fluid bed carbonizer and a lower pressure, circulating fluid bed combustor. The particle size distribution and density distribution from the carbonizer in an APFBC can be quite broad which promotes segregation, and in turn, leads to a loss in solids' flow to the combustor.

Segregation has been studied extensively in static fluid beds and evaluating the effects of bed materials and fluid bed geometry. Richardson and Zaki (1954) observed the tendency for different size glass spheres to segregate in liquid-solid fluidized bed. Rowe et al. (1972) characterized segregation of mixtures with particles of different solids densities in gas-solid fluidized beds. They were the first to refer to the material that settled to the bottom as jetsam and that which floated to the top as flotsam. For mixtures of solids with equal size but different densities the flotsam is made up of lighter material, while the jetsam is the heavier solids (Nienow at al., 1978; Nienow and Cheesman, 1980; Chiba et al., 1980; Naimer et al., 1982). For mixtures of solids with equal density but having either a wide size distribution or several discretely different size particles, the flotsam consists of the smaller and the jetsam the larger particles (Peeler and Huang, 1989; Nienow et al, 1987; Formisani, 1991; Wu and Baeyens, 1998). Mixing indices are defined based on the extent of segregation of different particles along the length of the fluid bed. Wen and Yu (1966) report that for a binary mixture a ratio of the minimum fluidization velocities greater than 2 was necessary for segregation. Rowe et al. (1972) found that in shallow beds the relative segregation in such mixtures is proportional to the ratio of the particle densities raised to the power 2.5 and to the ratio of particle diameters to the power of 0.2.

Shallow fluid beds, beds with low bed aspect ratios - D<sub>t</sub>/H<sub>bed</sub><0.8, were found to enhance segregation (Nienow et al., 1978; Wu and Baeyens, 1998). However, there are few studies of segregation in fluid beds having aspect ratios higher than D<sub>t</sub>/H<sub>bed</sub>>3 or in beds that are deeper than 1 m in height. Heights of 1 to 2 m are common in the loopseal of commercial CFB's; however, proposed

loopseal coolers and N-valve arrangement in the PFBC can be 4 to 30 m with aspect ratios from 8:1 to 150:1. Peeler and Huang (1989) studied large scales comprised of 0.61 and 1.22 m² fluid bed and 1.2 m high beds and found that a lower velocity was required for mixing than needed in the smaller cylindrical beds. They attributed this to the formation of much larger bubbles in the larger beds. The additional pressure exerted by a tall, fluidized column of solids is expected to increase the potential to segregate by reducing the size of bubbles. The same affect can be expected to arise in pressurized systems being proposed for advanced power systems.

The difference in the loopseal of CFB's and the segregation work reported in the literature is the fact that the fluid bed is static in the literature studies discussed above, but in the loopseal of a CFB, the solids are constantly moving in a "psuedo-plug flow" manner. This solids flux may provide a second mechanism for mixing in addition to bubbling. Under relatively low gas flow conditions the solids entering the bottom of the loopseal may displace those solids within the fluidized bed and force the bed upwards. This moving bed may reduce the extent to which segregation can occur in the non-mechanical valve. If solids are transported vertically up through the non-mechanical valve in a dense phase faster than the solids segregate, then segregation may be avoided. This paper will describe how the tests were performed, what factors most influence particle segregation in loopseals, and how these results may influence the design and operation of advanced pressurized fluid-bed systems.

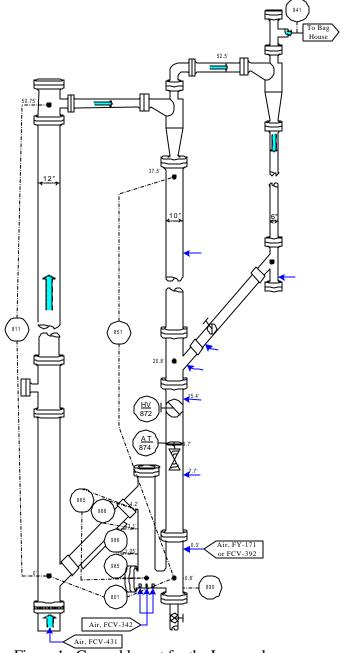


Figure 1. General layout for the Loopseal. installed in the CFB system showing key elevations and instrumentation.



Figure 2. Photograph of the base of the Loopseal looking horizontally along the length of the base with foreground being under the lift leg.

## **Experimental**

The loopseal configuration consisted of a 10-inch diameter and 5-foot high, fluidized bed lift-leg that was fluidized underneath through 7 aeration nozzles using flow loop FV-372 (Figure 1). The loopseal itself was fabricated from schedule 80 carbon steel and ½-inch thick acrylic pipe with ribbing reinforcement for ports where pressure differential and samples were taken. The transition from the standpipe to the lift-leg of the loopseal consisted of two short tees joined so the standpipe could be drained from

below through a 4-inch ball valve at its base. At the base of the lift-leg of the loopseal was a thick 10-inch diameter acrylic to permit inspection of the mixing caused by the fluidizing aeration below the lift-leg (Figure 2).

The solids circulating back into the CFB riser spill over the top of the loopseal and down the  $45^{\circ}$  slope and into the base of the riser. The main riser was 12-inch diameter and 51.5-feet tall. Primary riser air was supplied through a distributor plate consisting of many 1/8-inch holes. The riser velocity was operated at a sufficient velocity to maintain a dilute riser operating regime. When necessary mixing was accomplished within the riser by dropping the riser flow for a period of time and then abruptly blowing the solids over into the standpipe. To avoid any complication due to sample segregation or stratification within the unit the entire inventory of solids in the unit were cycledthrough the loop seal. By doing this any coarse materials with a tendency to segregate and become trapped within the loopseal will have an opportunity to do so. A minimum operation time was defined as being a single pass through the unit,  $t_1$ , such that:

$$t_1 = \frac{M_t}{M_s} \tag{1}$$

where  $M_I$  is the entire solids inventory in the unit, and  $\dot{M}_s$  is the solid circulation rate.

Gases and solids pass from the riser into a dual cyclone system each having greater than 99.9% efficiency. Solids from the primary cyclone drain into the 10 inch diameter standpipe or stand-leg and were moved into the loopseal using "move" aeration,  $F_m$  (FY-171), located 18 inches above the base of the standpipe.

Solids circulation rate was measured continuously using a 6-inch wide, helical-shaped vane having 180° twist over its 12-inch length (AT-874). This was located 10 feet above the base of the

standpipe. This spiral was suspended from a swivel and when the moving bed of solids flow past it the resulting rotations were counted by a 32-point digital encoder and was at a frequency of 0.5 Hz, using the bulk density, to the circulation rate. Differential pressure readings were taken along the axial location of the various components in the system at a frequency of 1 Hz.

Solids sampling was taken from 4 locations: 1) 2 feet above the bottom of the standpipe, 2) the bottom of the loop seal, and 3&4) 1.85 and 4.05 feet from the base of the lift-leg in the loop seal. In the loopseal solids sampling probes consisting of 3/8-inch s.s. tubing were extended into the center of the fluid bed. Because radial mixing is not a problem in the packed moving bed in the standpipe, standpipe samples were drained from a 3/4-inch port at the wall.

The solids densities were measured using a pycnometer with water as the fluid This method overestimates the densities for coke slightly. The particle size distribution was measured via ASTM sieve analysis methods. The mean surface area/volume, or Sauter mean diameter, was used to characterize the solids material size. Standard ultimate analysis and proximate analysis was used for C, H, ash, and moisture analysis of mixtures and individual bed components.

#### Results

A variety of solids were utilized, including various sizes of coke, quartz, and cork. A summary of the measured properties is represented in Table 1. The terminal velocity was calculated from standard drag relationships and particle properties. The size distribution for the test materials is presented in Figure 3. The particle sizes for coke ranged from 40 to 500 µm. and reflects a relatively wide size range compared to the quartz or various cork samples.

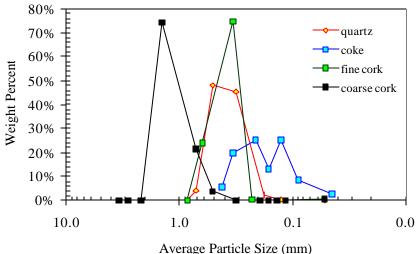


Figure 3. The size distribution for materials tested in the CFB loopseal system.

Minimum fluidization of various samples is presented in Figures 4 and 5. The minimum fluidization velocity is the intersection of the sloped line in the packed bed at low velocities and the plateau above the minimum fluidization. Note that for cork and coke samples that as the flow is increased the incremental  $\Delta P/L$  overshoots the maximum value until the packed bed is unlocked where bubbling occurs. For coke the bed is unlocked and bubbling occurs in the shorter 2.5-inch ID bed

near 0.11 ft/s (Figure 4). However, when the gas flow rate was increased in the 5-foot tall, loopseal, lift-leg, the velocity required to achieve complete fluidization was found to be 0.56 ft/s (Figure 5). During this latter tests there appears to be two linear slopes below fluidization, with the first transition near the 0.25 ft/s. Above this velocity the bed exhibits episodes of bubbling and stagnation causing fluctuations and the  $\Delta P$  increases gradually up to a maximum marking complete fluidization. Likewise, for the fine cork the minimum fluidization was 0.08 ft/s in the small (2.5-inch ID) fluid bed, but after circulated in the fluid bed and tested in the loopseal the minimum fluidization velocity was much higher, 0.28 ft/s.

Table 1. Relevant characteristics of test materials.

|                            |       |        |       | 30x40 | 20x30 | 60x70 | 20x50 |
|----------------------------|-------|--------|-------|-------|-------|-------|-------|
|                            | units | quartz | coke  | coke  | coke  | cork  | cork  |
| Physical properties:       |       |        |       |       |       |       |       |
| $\mathbf{d}_{\mathbf{p}}$  | μm    | 385    | 202   | 500   | 707   | 356   | 1032  |
| $\mathbf{r}_{\mathrm{b}}$  | g/cc  | 1.35   | 0.87  | 0.76  | 0.75  | 0.056 | 0.103 |
| $\mathbf{r}_{\mathrm{vb}}$ | g/cc  | 1.57   | 0.88  | 0.83  | 0.81  | 0.068 | 0.090 |
| $\mathbf{r}_{\mathrm{s}}$  | g/cc  | 2.66   | 1.71  | 1.69  | 1.89  | 0.14  | 0.19  |
| r <sub>He</sub>            | g/cc  | NA     | 1.92  | NA    | 0.63  | 0.81  | 0.58  |
| Hydrodynamic properties:   |       |        |       |       |       |       |       |
| $\mathbf{u}_{\mathbf{mf}}$ | ft/s  | 0.59   | 0.08  | 0.57  | 0.90  | 0.08  | 0.56  |
| $\mathbf{u_{cf}}$          | ft/s  | NA     | 0.56  | NA    | NA    | 0.28  | NA    |
| $\mathbf{u_t}$             | ft/s  | 2.06   | 0.45  | 2.14  | 3.10  | 0.10  | 0.97  |
| $\mathbf{e}_{vb}$          |       | 0.41   | 0.50  | 0.51  | 0.51  | 0.51  | 0.45  |
| $\mathbf{e}_{mf}$          |       | 0.45   | 0.56  | 0.54  | 0.54  | 0.54  | 0.51  |
| Chemical Analysis:         |       |        |       |       |       |       |       |
| moisture                   | wt. % | 0.00   | 0.57  | NA    | NA    | NA    | NA    |
| ash                        | wt. % | 100.00 | 8.53  | NA    | NA    | NA    | NA    |
| H                          | wt. % | 0.00   | 0.61  | NA    | NA    | NA    | NA    |
| $\mathbf{C}$               | wt. % | 0.00   | 89.66 | NA    | NA    | NA    | NA    |

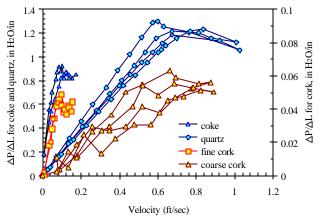


Figure 4. The minimum fluidization velocity for test materials as measured with increasing velocities in the 2.5-inch ID static fluid bed.

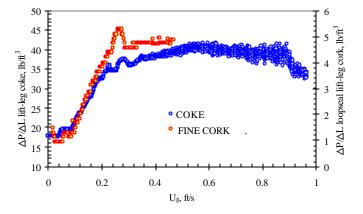


Figure 5. Fluidization curve taken in 5' high loopseal lift-leg with increasing velocity for fine cork and for coke.

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Several factors contribute to these observations including differences in the size distribution and the bed height. Richardson and Zaki (1954) report that the minimum fluidization velocity is dramatically reduced with the addition of fines. Just a small amount of fines fill interstitial voids between larger particles and promote fluidization. In the loopseal tests, prior to testing, the coke was circulated around the CFB and the cyclone separators in the CFB unit are quite efficient at removing fine particulate. Gauthier at al. (1999) report similar behavior during fluidization tests for binary mixtures and for samples with wide particles size distributions. In other words, narrow and Gaussian particle distributions exhibit well defined packed bed and fluid bed regions in the minimum fluidization curve. But samples with wide size distributions and/or bimodal size distributions exhibit a third region between packed bed and complete fluidization in which there is a more shallow rise in bed  $\Delta P$  versus gas flow. A similar response was observed with coke in the loop seal. Additional flow is required above minimum fluidization to achieve complete fluidization depending upon the extent of compaction in the bed. Greater compaction results in higher flow needed to achieve complete fluidization. The effect of a higher bed height in the loop seal results in higher compaction. This viewpoint explains why velocities of several times the minimum fluidization were necessary to get complete fluidization.

Coke with wide size distribution. Tests were conducted at low solids flow rate indicative of APFBC operations using gas flows of  $1.5 \cdot U_{mf}$   $2 \cdot U_{mf}$  and  $3 \cdot U_{mf}$  (Table 2). The loopseal apparently was reduced slightly in the concentration of fines at the bottom after about 1 hour of operation. This segregation was greater at the lower fluidization velocity. The first test condition was established only after mixing the bed in the loopseal holding a velocity of  $5 \cdot U_{mf}$  for a period of 10 minutes and circulating solids through the loopseal. At that time the fluidization velocity in the loopseal was reduced to about  $1.5 \cdot U_{mf}$ . This reduction in flow, however, resulted in a loss in solids circulation rate. Flow could not reestablished again until fluidization velocity was approximately twice  $U_{mf}$ . The second condition was fully established when the solids circulation rate was brought back down to the desired 1,400 lb/hr. The solids inventory in the loopseal was being turned over about every 5 minutes at these solids flow rates.

Table 2 is a summary of size analyses for the coke tests. A segregation ratio,  $C_{\rm s}$ , can be defined as:

$$C_{s} = \frac{(X_{b} - X_{t})}{(X_{b} + X_{t}) \times 100}$$
 (2)

where  $X_b$  is the concentration of the segregating material in the bottom of the fluid bed, and  $X_t$  is its concentration in the top of the fluid bed. From this equation the segregation coefficient,  $C_s$ , can have a value between -100 and +100 with a value of 0 indicating perfect mixing (Geldart, et al., 1981). Particle size was used in this equations in two ways: either based upon either the mean particle sizes or the weight % material larger than 420 microns. Since volumetric concentrations were desired to evaluate mixing, the mean size was taken as  $d_p^3$  and substituted for concentrations. However, the use of the coarse size cut (wt %) was a more meaningful measure of the jetsam. This latter segregation coefficient indicated that mixing was best ( $C_s \sim 0$ ) at higher velocity and decreased as velocity decreased. The key process finding was that segregation occurred while the flow was still well above minimum fluidization, namely 1.5 to  $2 \cdot U_{mf}$ .

| Table 2. | Segregation | coefficients | for tests | with co | ke breeze | (9/17/99). |
|----------|-------------|--------------|-----------|---------|-----------|------------|
|          |             |              |           |         |           |            |

|                               | d <sub>p</sub> , Sauter mean      |     |     |       | Wt.% >420 um |        |     |                      |
|-------------------------------|-----------------------------------|-----|-----|-------|--------------|--------|-----|----------------------|
| <b>Test Condition</b>         | Stand-leg Bottom Top $C_s(d_p^3)$ |     |     |       | Stand-leg    | Bottom | Top | C <sub>s</sub> (wt%) |
| Initial after static          | 214                               | 175 | 149 | 24.1  | 39           | 36     | 31  | 21.9                 |
| fluidization test             |                                   |     |     |       |              |        |     |                      |
| 3·U <sub>mf</sub> 1,400 lb/hr | 201                               | 205 | 180 | 19.6  | 36           | 37     | 37  | 2.0                  |
| 3·U <sub>mf</sub> 1,400 lb/hr | 229                               | 207 | 193 | 10.8  | 42           | 39     | 37  | 7.1                  |
| 2·U <sub>mf</sub> 1,400 lb/hr | 246                               | 193 | 207 | -10.6 | 43           | 36     | 41  | -20.1                |
| 1.5·U <sub>mf</sub>           | *                                 | *   | *   | *     | *            | *      | *   | *                    |

<sup>\*</sup>Operations were unstable since circulation rate could not be maintained. Samples were not obtained.

The variability in size due to attrition and axial stratification in the standpipe complicated analysis of segregation using the mean size analysis. For example, the initial sample was taken after the minimum fluidization test. This sample had markedly lower mean particle size than any of the samples after circulation was established. The fact that both the bottom and top samples were smaller in size than the standpipe samples suggests that attrition was occurring, most likely near the nozzles at the base of the loopseal riser. Batches of fines were also evident when fines intermittently moved from the secondary solids separator (via the dipleg) into the upper region of the moving standpipe bed. Varying the operations in the system did not readily eliminate such axial variations; however, when the entire inventory of solids was cycled through the loopseal segregation was observed in the coarse size cut.

To evaluate axial stratification in the bed, samples were taken as a function of time for a given condition. Measurements along the standpipe over a period of testing with coke produced as wide a variability as that observed along the axis of the loopseal itself. The coarsest material, however, did tend to segregate out into the bottom of the lift-leg of the loopseal when conditions were favorable. As long as the bed inventory was cycled around the system more than once, segregation was observed in the  $d_p>420~\mu m$  size fraction.

To test the effects of solids circulation rate the loopseal fluidization flows through the loopseal were varied from 1.2 to 3.8 times the minimum fluidization velocity and solids circulation rate varied from 4900, 32000, 39000, and 45000 lb/hr. The total  $\Delta P$  across the lift-leg of the loopseal was found to be insensitive to mass circulation rate and loopseal fluidization flow above the minimum fluidization velocity of the coke. The loopseal demonstrated stable performance, with invariant  $\Delta P$  across the loopseal of 1.75 psid, over this range of operating flows while using varied standpipe aeration schemes.

**Coarse and fine cork**. A 50:50 mixture of 20x50 and 60x70 mesh cork was circulated for an extended period of time at a relatively high circulation rate. No evidence of segregation was detected by following the differential pressure measurements within the loopseal. However, eventually the loopseal stopped bubbling/fluidizing and the circulation rate dropped to zero over a relatively short

period of time. As was the case with the coke of wide size distribution, increasing the fluidizing airflow rate was necessary to re-establish the solids circulation. The amount of increase required corresponded to an increase from 0.5 to  $2~U_{\rm mf}$  of the coarse cork component.

Coke and quartz. Tests were conducted with a mixture of coke and quartz. The primary objective of the tests was to determine whether the quartz could be circulated through the loopseal. These tests explored the dependence of fluidizing airflow and system pressure on loopseal performance such as mass circulation,  $\Delta P$  across the loopseal, and segregation of solids. We did this by draining bed material from the bottom of the loopseal, pouring the coke onto the top of the lowered bed level, and then replacing the drained coke back onto the top of the bed. Four steady state operating periods were achieved with all flows being cut-off in between each to pull samples and add quartz, LT1 through LT4A in sequence. Before each of these tests, 10 lb. of quartz was added into the middle of the loopseal. The unit contained about 900 lb of coke. Test results are summarized in Table 3 and 4. The final operating period included a series of gradually decreasing fluidizing airflows until solids circulation became unreliable.

Table 3. Operating conditions and results (5 min. average values) for tests with coke and quartz mixtures in 10 lb. of quartz were added to the loopseal prior to each of the following cases: LT1, LT2, LT3, and LT4a.

| Tag No.                       |                     | CASE:              | <u>LT1</u> | <u>LT2</u> | <u>LT3</u> | LT4a  | LT4b  | LT4c  | LT4d  | LT4e  |
|-------------------------------|---------------------|--------------------|------------|------------|------------|-------|-------|-------|-------|-------|
| Msr'd Par.                    | <b>Description</b>  | <u>units</u>       |            |            |            |       |       |       |       |       |
| PDT-865 LPSI                  | L BOT - TOP         | PSID               | 1.49       | 1.71       | 1.75       | 1.81  | 1.83  | 1.86  | 1.88  | 2.00  |
| PDT-985 LPSI                  | L -00.80 - 01.05    | lb/ft <sup>3</sup> | 49.8       | 51.6       | 53.2       | 52.6  | 53.7  | 55.1  | 53.3  | 54.8  |
| PDT-986 LPSI                  | L 01.05 - 03.10     | lb/ft <sup>3</sup> | 46.5       | 52.4       | 51.3       | 51.2  | 52.3  | 51.7  | 53.6  | 58.3  |
| PDT-988 LPSI                  | L 03.10 - 04.00     | lb/ft <sup>3</sup> | 27.1       | 42.3       | 43.7       | 47.8  | 48.4  | 48.9  | 49.7  | 51.0  |
| PT-890 SP B                   | OT                  | PSIG               | 1.369      | 1.584      | 6.159      | 6.205 | 6.132 | 8.055 | 8.699 | 8.935 |
| $AT-874 M_s$ , s              | spiral              | Lb/hr              | 2742       | 2702       | 1910       | 2899  | 3024  | 3133  | 2976  | 2993  |
| FT-342 LPSI                   | L FLZN              | SCFH               | 1301       | 600        | 850        | 550   | 500   | 451   | 402   | 350   |
| Calc'd Par.:                  |                     |                    |            |            |            |       |       |       |       |       |
| $U_{g}$ Loo <sub>l</sub>      | oseal velocity      | ft/s               | 0.724      | 0.333      | 0.325      | 0.210 | 0.193 | 0.172 | 0.151 | 0.134 |
| ρ <sub>g</sub> Air o          | density @ rsr bot   | Lb/ft <sup>3</sup> | 0.083      | 0.082      | 0.119      | 0.119 | 0.118 | 0.119 | 0.122 | 0.119 |
| U/U <sub>MF quartz</sub> Rela | tive flzn. velocity |                    | 1.24       | 0.57       | 0.55       | 0.36  | 0.33  | 0.29  | 0.26  | 0.23  |

The pressure profile across the loopseal displayed little relative variation, were consistent, and repeatable. The differential pressure across the top of the lift-leg increased systematically as the flow decreased and pressure increased. Solids circulation also was relatively smooth and stable until the loopseal velocity was dropped to below twice the minimum fluidization velocity of the finer coke material, case LT4d. For these low flow cases the solids circulation became sporadic. The loopseal fluidization would cease, gas pressure would build under the loopseal, followed by surging and bubbling once sufficient gas pressure had built there to reinitiate fluidization in the loopseal. This was characterized by increased loopseal pressure fluctuations.

Table 4. The concentration of quartz (wt. %) and estimated mixing indices in the loopseal lift-leg with coke and quartz mixtures at various flows and pressures.

| test no.                          | LT1  | LT2  | LT3  | LT4C |
|-----------------------------------|------|------|------|------|
| wt.% quartz                       | 1.54 | 3.08 | 4.62 | 6.15 |
| P, psig                           | 1.37 | 1.58 | 6.16 | 8.05 |
| U/U <sub>mf quartz</sub>          | 1.24 | 0.56 | 0.55 | 0.29 |
| Quartz conc. Wt% Sample location: |      |      |      |      |
| Stand-leg @1.6'                   | 0.90 | 6.60 | 5.29 | 6.14 |
| Lift-leg @ 0'                     | 5.08 | 5.95 | 4.71 | 6.06 |
| Lift-leg @ 1.3'                   | 5.82 | 5.28 | 6.00 | 5.00 |
| Lift-leg @ 4'                     | 6.42 | 1.44 | 0.90 | 1.55 |
| Avg. x <sub>j</sub>               | 5.8  | 4.2  | 3.9  | 4.2  |
| $C_{\rm s}$                       | 12   | -61  | -68  | -59  |
| $\mathrm{M}_{\mathrm{p}}$         | 1.11 | 0.34 | 0.23 | 0.37 |

The highest quartz concentration was observed in the top of the loopseal for LT1 with  $U/U_{mf}$  quartz=1.24. This case also had the lowest  $\Delta P/\Delta L$ , bulk densities (59-61 lb/ft³) and the smallest mean size particles (190-200  $\mu$ m). The middle and bottom portions of the bed had higher  $\Delta P/\Delta L$ , bulk densities (63-65 lb/ft³), and particle sizes (220-250  $\mu$ m except one low value at the bottom). The upper bed can most easily expand and expel solids at high velocities resulting in higher voidages and lower local bed densities. This portion of the bed most readily responded to changes in flow. The high gas flow was obviously enough to carry quartz up and over to the standpipe as evident by the concentrations of quartz observed. For the low flow cases the increased bed density in the bottom and middle of the loopseal resulted mainly from segregation illustrated by increasing quartz concentrations. The pressure drop across the bottom of the loopseal increased slightly with flow.

The increased bulk densities in all these samples as compared to coke (~54 lb/ft3) supports this measured enrichment of quartz everywhere in the unit. Most notably the quartz analysis of the standpipe samples indicated that sand has in fact been circulated through the loopseal. This is consistent with visual observation that white sand could be seen in the standpipe for all of these operating conditions.

Lower loopseal airflows, those between minimum fluidization of the coke and quartz, produced samples with higher bulk densities. A segregation coefficient,  $C_s$ , was calculated based upon the carbon or ash analyses by substituting the following into eq. 2 above:

$$X_{\text{quartz}} = (C_{\text{mix}} - C_{\text{coke}})/(C_{\text{quartz}} - C_{\text{coke}}) * 100$$
(3)

where  $C_{mix}$  is the concentration of carbon in the sample, and  $C_{quartz}$  and  $C_{coke}$  are the carbon contents in the original quartz and coke component, respectively. A comparison of carbon against using ash content provided excellent agreement for coke and quartz mixtures. In situations where sampling was not conducted, a mixing index,  $M_p$ , based upon  $\Delta P$  data was used. This index is defined as:

$$M_{p} = \Delta P_{bot} - \Delta P_{top} / \Delta P_{loopseal}$$
 (4)

where  $\Delta P_{bot}$  is the pressure drop per unit length at the bottom of the loopseal,  $\Delta P_{top}$  the pressure drop per unit length at the top of the loopseal, and  $\Delta P_{loopseal}$  the pressure drop per unit length across the entire length of the loopseal. The mixing index approaches zero when there is poor, and unity when there is good mixing.

Both of the estimated mixing indices indicated better mixing for the flow above the minimum fluidization velocity for the quartz jetsam and segregation for lower flows. The higher pressure was found to be only slightly less well mixed than the atmospheric case. This trend however was consistent with the concept that higher pressure produces smaller bubbles and thus a greater tendency to segregate (Rowe et al., 1972). Unfortunately, the value of  $M_p$  can be misleading low if the denser solids are no longer fluidized but become static.

Coke and cork. Tests of mixtures of coke and cork were evaluated in a fashion similar to that of the coke and quartz. The mixture consisted of 125 lb. of 20x50 mesh cork and 10 lb. of 30x40 mesh coke loaded to the CFB unit. The fluidization velocity and the circulation rates were varied to evaluate the affects of each on mixing (Figure 6-8). The lift-leg pressure drop decreased as the velocity exceeded that required to fluidize the coke material until the  $\Delta P/\Delta L$  level was that obtained for the cork itself (Figure 6). This indicated that the more dense coke was being circulated and dispersed throughout the entire CFB when the flow was sufficient.

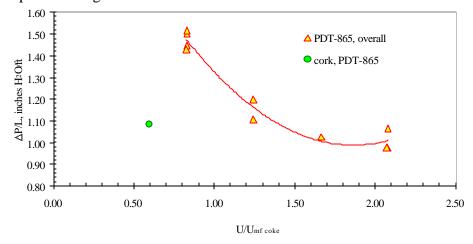


Figure 6. The effect of fluidization velocity on the  $\Delta P$  across the lift-leg of the loopseal for a12.5:1 wt. mixture of 20x50 mesh cork and 30x40 mesh coke.

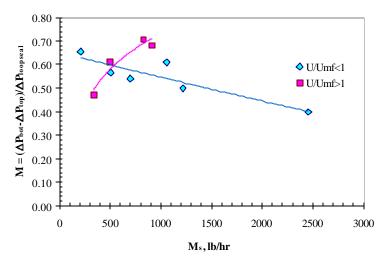
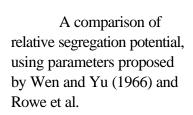


Figure 7. The effect of solids circulation rate on mixing as measured by differential pressures across the lift-leg of the loopseal for a12.5:1 wt. mixture of 20x50 mesh cork and 30x40 mesh coke.

The mixing index estimated from the  $\Delta P$  across the lift-leg suggests fairly good mixing for all condition with values of M<sub>p</sub> between 0.45 and 0.75. When the circulation rate was increased, within the range of expected solids flows for an APFBC, this mixing indicator depended upon whether the fluidization flow was above or below the velocity required to fluidize the jetsam coke material (Figure 7). Below  $U/U_{mf, coke}$  the value for M<sub>p</sub> tended to decrease slightly with increased circulation rate, while above U/U<sub>mf. coke</sub> the opposite was observed. The improved mixing

obtained in the latter case, however, was likely due to the increased fluidization velocity since this variation was much larger than that for the circulation rates (Figure 8).

Analysis of samples for coke concentrations is presented in Table 5. Apparently the denser material did circulate to the standpipe, but not for all cases. The data do not reflect a simple relationship with fluidizing velocity since the best mixing index was found in intermediate flow conditions.



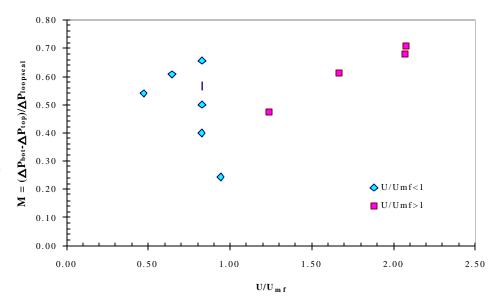


Figure 8. The effect of fluidization velocity on mixing as measured by differential pressures across the lift-leg of the loopseal for a 12.5:1 wt. mixture of 20x50 mesh cork and 30x40 mesh coke.

Table 5. Concentrations of coke (wt. %) and estimated mixing indices for the loopseal lift-leg with 10 lb. coke (30x40) added to 125 lb. cork (20x50) mixtures at various flows and pressures.

|                           | L14   | L12   | L13   | L17   |
|---------------------------|-------|-------|-------|-------|
| U/U <sub>cf</sub>         | 0.83  | 1.24  | 1.67  | 2.07  |
| Coke conc. Wt%            |       |       |       |       |
| sample location:          |       |       |       |       |
| stand-leg @1.6'           | -2.00 | 3.43  | 1.58  | -1.06 |
| lift-leg @ 0'             | 72.66 | 16.60 | 37.58 | 24.14 |
| lift-leg @ 1.3'           | 5.78  | 9.72  | 8.27  | 7.87  |
| lift-leg @ 4'             | 4.28  | 24.23 | 54.34 | 8.25  |
| avg.x <sub>j</sub>        | 28    | 17    | 33    | 13    |
|                           |       |       |       |       |
| $C_s$                     | -89   | 19    | 18    | -49   |
| $\mathrm{M}_{\mathrm{p}}$ | 0.16  | 1.44  | 1.63  | 0.61  |

Table 6. Comparison of the segregation potential for various mixtures tested.

|   | coarse/fine coarse/fin |      | 20x50 cork  |            |           |  |
|---|------------------------|------|-------------|------------|-----------|--|
|   | Coke                   | cork | quartz/coke | 30x40 coke | coke/cork |  |
| Segregation potential (>2):                           |                        |      |             |            | _         |  |
| $\mathbf{u}_{\mathrm{mf1}}/\mathbf{u}_{\mathrm{mf2}}$ | 18.00                  | 7.30 | 7.66        | 1.35       | 1.01      |  |
| Relative segregation:                                 |                        |      |             |            |           |  |
| $\mathbf{r}_{1}/\mathbf{r}_{2}$                       | 1.14                   | 1.35 | 1.55        | 8.95       | 12.21     |  |
| $\mathbf{d_1}/\mathbf{d_2}$                           | 7.86                   | 2.90 | 1.91        | 0.48       | 0.57      |  |
| $(\mathbf{r}_1/\mathbf{r}_2)^{2.5}$                   | 1.38                   | 2.11 | 3.01        | 239.82     | 521.40    |  |
| $(\mathbf{d_1}/\mathbf{d_2})^{0.2}$                   | 1.51                   | 1.24 | 1.14        | 0.87       | 0.89      |  |

(1972), for the various mixtures tested helps to shed some light on these results (Table 6). According to Wen and Yu's criteria the equi-density mixtures and quartz/coke mixtures had potential to segregate, but neither of the coke/cork mixtures studied would have been expected to segregate. The varied results found for the coke/cork mixtures could reflect the tendency for the solids to invert or may simply reflect the general variability of a two components circulating around a CFB loop. We however did observe several occasions of segregation and purging of jetsam from the lift-leg (Figures 9-10).

Rowe's relative segregation parameters were mostly consistent with Wen and Yu's segregation potential criteria. The equi-density and quartz/coke mixtures had diameter and density parameters all greater than 1 predicting a consistent tendency favoring segregation. For the coke/cork mixtures these parameters varied in opposite directions and as such were consistent with the low segregation potential. However, the magnitude of the density parameter for this mixture was 2 orders of magnitude larger than any of the other parameter estimates. By comparison the diameter parameter was only 15% on the low side. Thus, these measurements of relative segregation supported the observation that segregation occurs in the coke/cork mixtures.

Recovery and Purge Methods. Evidence of segregation was observed with both the density and size mixtures. This segregation, however, was found to be reversible by simply increasing the aeration rates in the loopseal riser to a flow that was above the minimum fluidization velocity of the top size for the densest particles (Figures 9 and 10). Purge methods were evaluated and surge events were minimized using a gradual increase in aeration rates. When the fluidizing velocity was increased rapidly by changing the set-point to a higher airflow, the loopseal was purged of the jetsam, but several undesirable events occurred. Increasing the flow reduced the solids inventory in the lift-leg, and so the solids flow out of the loopseal increased for a short period of time (Figure 9). This was evident by a large spike in the  $\Delta P$  measured over the main riser of the CFB. The pressure drop across the lift-leg also decreased reflecting the removal of the denser jetsam. A slower airflow ramp was successfully used several times over similar flow conditions for this same mixture (Figure 10). This resulted in

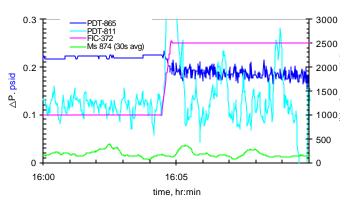


Figure 9. Loopseal purge using rapid increase in fluidization airflow from 0.8 to 2 times  $u_{\rm mf}$  of coke. Bed material consisted of 20x50 mesh cork and 30x40 coke (2/10/00).

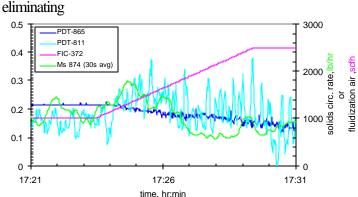


Figure 10. Loopseal purge using a ramped increase in fluidization airflow from 0.8 to 2 times  $u_{\rm mf}$  of coke over a 6 minutes period. Bed material consisted of 20x50 mesh cork and 30x40 coke (2/10/00).

the spike of solids flowing out of the loopseal while the circulation rate was maintained fairly constant.

## **Conclusions**

A 5-foot high and 10-inch ID Loopseal was operated under ambient conditions to evaluate segregation. Experiments within this loopseal were conducted with coke, cork, and quartz in various size and density mixtures. The effects of fluidizing air to the loopseal, mass circulation rate, and solids' mixture on segregation were evaluated. Evidence of segregation was observed with both the density and size mixtures.

Mixing indices were used to evaluate the effects of fluidizing gas velocity, pressure, and solids

circulation rate. Measurements using pressure differentials were useful only for testing mixtures of different densities. In addition, when the fluidization rates were below the minimum fluidization velocity of one of the components, the pressure differential method could produce an erroneously high mixing index. Sampling solids for size and concentrations required multiple samples due to axial stratification throughout the CFB loop. When this was done physical and chemical analyses were equally successful for estimating concentrations of components in various density mixtures. Size distribution in the CFB loop tested here proved to be more problematic due to the wide variations observed and the lack of understanding of the causes and effects of operational changes on size variations. However, segregation coefficients based upon the amount of the coarsest size cut were found to be relatively useful in identifying conditions that promote or avoid segregation.

Fluidization measurements in different size beds were used to identify the required flow to avoid particle segregation. For mixtures with a tendency to segregate, mixing could be achieved by utilizing gas flows sufficient to fluidize the jetsam. As expected from theory that bubbles in a fluid bed promote mixing, it was observed that higher pressure and/or deeper bed depths were deleterious to mixing. Deeper beds exhibit greater compaction resulting in higher velocity required to achieve complete fluidization. The extent of this velocity increase was measured by comparing fluidization plots from a small shallow fluid bed to those measured in-situ in the 10-inch diameter, 5-foot tall loopseal. In a shallow bed minimum fluidization is measured, but if the bed material consists of a wide particle size distribution or if there are different density components, then a deep bed also exhibits a higher velocity transition representing "complete" fluidization.

Test comparing mixing indices for a variety of mixtures demonstrated that the flow required to avoid segregation within the deeper loopseal bed was above the minimum fluidization velocity of the jetsam. In an equi-density mixture this is synonymous to maintaining a flow above the measured flow at "complete" fluidization. It is recommended to stay above twice the minimum fluidization of the jetsam to avoid segregation. However, when complete fluidization velocities can be determined in the process vessel of interest then a margins as small as 25% above the complete fluidization were used without destabilizing segregation occurring.

Segregation was the greatest when operating the loopseal at fluidizing velocities between that of the minimum fluidization velocities for the flotsam and jetsam components. The mixing can be thought to be aggravated in this situation because the flotsam is mobilized and thereby can be separated from the jetsam. The jetsam is not suspended under these conditions and will settle to the bottom.

Higher pressures, on the other hand, reduce the size of bubble in the fluid bed and thereby reduce the extent of mixing resulting from the rise of the bubbles through the bed. Data suggests that while this may happen the extent of this effect is small. Further work is needed in this area.

The flow of solids through the loopseal did promote mixing, but rates of solids flow sufficient to

eliminate segregation were larger than those proposed for the APFBC applications. Some evidence was found that increasing the solids circulation rate improved mixing in the coke/cork mixtures. High solids flow rates were quite successful in maintaining a stable smoothly operating loopseal when operated with coke having a wide particle size distribution. This was true when operating as low a flow into the loopseal as 1.2 times minimum fluidization for the coke. The extent of this effect still needs to be determined.

Segregation was found to be reversible by simply increasing the aeration rates in the loopseal riser to a flow that was above the minimum fluidization velocity of the top size for the densest particles. Purge methods were evaluated and surge events were minimized using a gradual increase in aeration rates. Application of existing correlations to evaluate segregation potential provided insight to interpreting test results.

# **List of Symbols**

 $\begin{array}{ll} C_{mix} & \text{concentration of carbon in the mixture, wt. \%} \\ C_{coke} & \text{concentration of carbon in the coke, wt. \%} \\ C_{quartz} & \text{concentration of carbon in the quartz, wt. \%} \end{array}$ 

C<sub>s</sub> segregation ratio, dimensionless

d diameter, ft

D vessel diameter, ft d<sub>p</sub> particle diameter, μm

F<sub>m</sub> "move" aeration near base of the standpipe, scfh

L Length, ft

M<sub>I</sub> solids inventory in the CFB unitM mixing index, dimensionless

 $M_p$  mixing index based on incremental  $\Delta P$ 's across the fluid bed, dimensionless

*M*<sub>s</sub> solids circulation rate, lb/hr

P pressure, psig

t<sub>1</sub> cycle time for solids to pass once around the CFB loop, hr

 $\begin{array}{ll} U & \text{superficial gas velocity, ft/s} \\ u_{mf} & \text{minimum fluidization velocity, ft/s} \\ u_{cf} & \text{complete fluidization velocity, ft/s} \\ \end{array}$ 

u<sub>t</sub> terminal velocity, ft/s

X<sub>b</sub> concentration of jetsam in the bottom of the fluid bed, %

 $x_{j}$  concentration of the jetsam in the fluid bed, %  $X_{\rm ouartz}$  concentration of quartz in the fluid bed, %

 $X_t$  concentration of tracer property in the top of the fluid bed, %

### **Greek letters**

 $\varepsilon_{mf}$  void fraction at minimum fluidization

 $\varepsilon_{vb}$  void fraction at vibrated packed bed

 $\Delta L$  incremental length, ft

 $\Delta P$  pressure drop, psid

 $\Delta P/\Delta L$  bed density, lb/ft<sup>3</sup> or inches H<sub>2</sub>O/ft<sup>3</sup>

ρ density, lb/ft<sup>3</sup>

 $\rho_b$  bulk density, lb/ft<sup>3</sup>

 $\rho_{\rm g}$  gas density, lb/ft<sup>3</sup>

 $\rho_{vb}$  vibrated packed bed density, lb/ft<sup>3</sup>

 $\rho_s$  solids particle density, lb/ft<sup>3</sup>

ρ<sub>He</sub> solids density measured using helium B.E.T. adsorption, lb/ft<sup>3</sup>

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